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EFFECT OF VARIOUS FACTORS ON THE CORROSION AND RUSTING OF TOOLING MATERIAL USED FOR TABLET MANUFACTURING

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ABSTRACT

Various factors that cause the rusting of tablet punches and dies by a hydrochloride salt were evaluated. Tooling material rich in nickel content was found to have the best resistance to rusting by the hydrochloride salt. Other factors such as humidity, temperature and contact time with this hydrochloride salt were also found to be responsible for rusting of tooling material.

The hydrogen chloride liberated from the salt was found to be the cause of rusting of tooling. A correlation between the stability of hydrochloride salt as determined by thermogravimetric analysis and its effect on rusting of tooling material was demonstrated. Optimization of tooling composition during preformulation is recommended.

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INTRODUCTION

The rusting of punches and dies during compaction of certain granules is a continuing concern in the pharmaceutical industry[1]. Rust and its accompanying pitting on the punch cup could result in picking and obliteration of any delicate engraving. The use of poor quality tools can result in work stoppage as well as poor quality products.

In the current studies, we evaluated various factors that contribute to the rusting of tooling materials in contact with a hydrochloride salt. The factors include composition of tooling material, humidity condition, temperature and the contact time between a hydrochloride salt and tooling materials.

MATERIALS AND METHODS

Materials

REV 6000-A(SS), a proprietary hydrochloride salt developed by Revlon Health Care Group, was used. Various tooling materials as shown in Table 1[2] were used for visual observation of rusting.

Methods

The thermogravimetric analysis (TGA) of REV 6000-A(SS) was performed with a DuPont 951 thermogravimetric analyzer with a 990 thermal analyzer. The material was heated at a rate of 5°C/min. in an atmosphere of nitrogen.

TABLE 1

COMPOSITION OF VARIOUS TOOLING MATERIALS

Steel Analysis Nominal Composition (%)	Tool Designation				
	D2	D3*	DM	S5**	440C***
Carbon	1.40- 1.60	2.10- 2.30	0.50	0.50-0.60	0.95-1.20
Manganese	0.20- 0.40	0.20- 0.40	0.50	0.75-1.00	1.0
Silicon	0.20- 0.40	0.10- 0.40	0.25	1.75-2.25	1.0
Chromium	11.50-12.50	11.50-12.50	0.75	0.15-0.40	16.0-18.0
Vanadium	0.20- 1.00	0.20- 1.00	-	0.15-0.30	-
Molybdenum	0.70- 0.90	0.70- 0.90	-	0.20-0.50	0.75
Nickel	-	0.50	3.00	-	0.50
Sulfur	0.12	-	-	0.12	-

* Composition normally used in dies for commercial tablet production

** Composition normally used in punches for commercial tablet production

*** Stainless steel

REV 6000-A(SS) powder was stored in contact with various tooling materials up to 24 hours at 22°C/30% R.H., 22°C/85% R.H. and 35°C/85% R.H. This experiment was repeated by using a wet mass of powder with 10% water at 22°C/30% R.H. A scale of 0-3 was used for rating the degree of rusting, zero indicating no rusting. All the observations were made by the same individual and at the same time.

RESULTS AND DISCUSSION

Thermogravimetric Analysis (TGA)

The TGA of REV 6000-A(SS) (Figure 1) indicated a 9% weight loss occurring between 147° to 175°C. This corresponds roughly to a mole of hydrogen chloride. REV 6000-A(SS) has a melting point of 147°C which indicates that the liberation of HCl occurs on melting. There was, however, a rapid and continuous weight loss above 200°C due to decomposition of REV 6000-A(SS). The gas evolved on heating REV 6000-A(SS) powder in a test tube at 150° to 160°C was confirmed to be hydrogen chloride by passing the vapor over concentrated ammonium hydroxide. A pH of less than 1 was indicated by exposing wet litmus paper to the evolved gas.

Effect of Dry Powder on Tooling Material at 22°C/30% R.H.

The data as presented in Table 2 indicate that the REV 6000-A(SS) dry powder causes rusting of all tooling materials which was found to increase with time. These results seem to be correlated with the release of hydrogen chloride from REV 6000-A(SS) as dmonstrated by TGA (Figure 1).

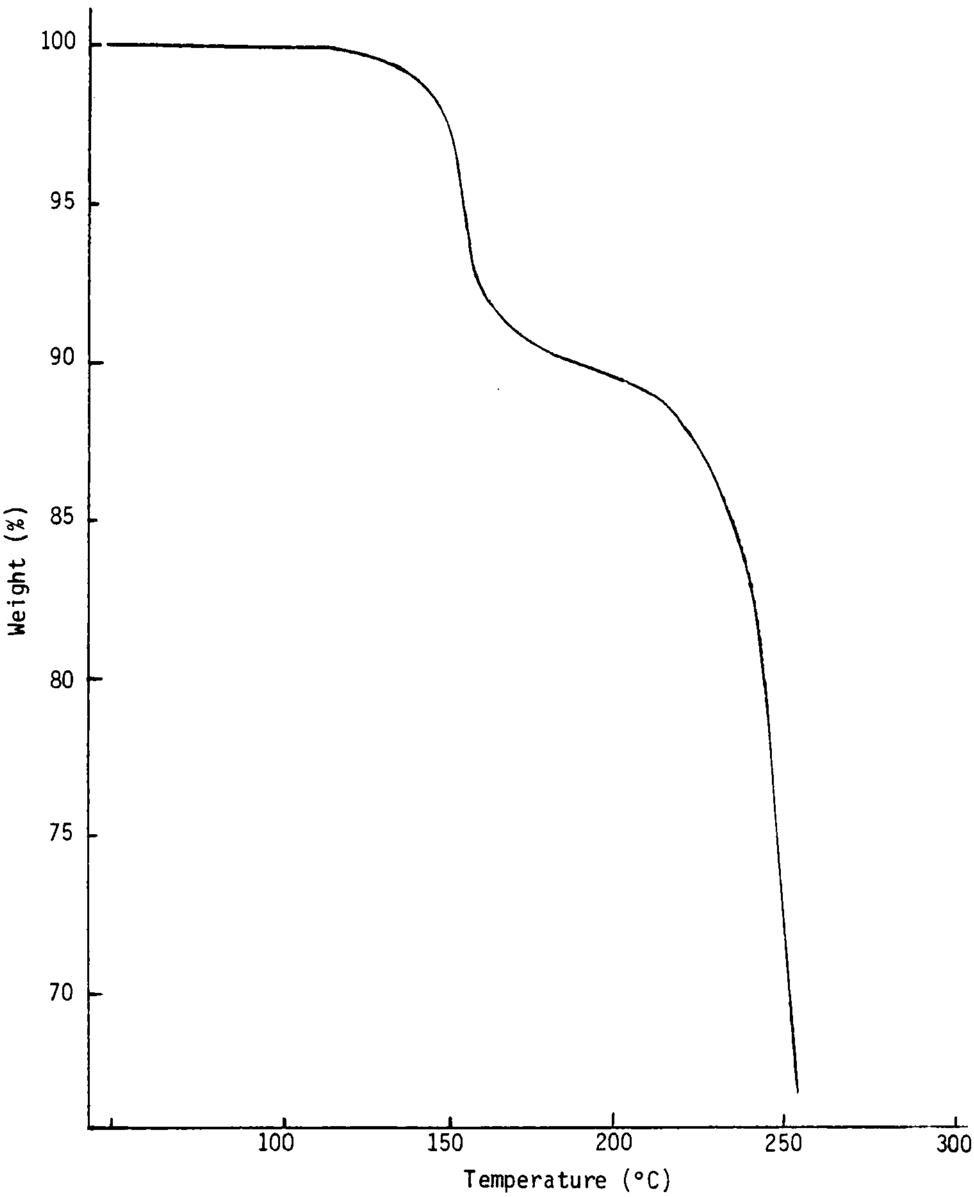

Figure 1: Thermogravimetric Analysis of REV 6000-A(SS)

TABLE 2

EFFECT OF DRY POWDER ON TOOLING MATERIAL
22°C/30% R.H.

Tooling Material	Rusting (Rating*)	
	2 Hours	24 Hours
D2	0	2
D3	0	2
DM	0	1
S5	1	3
440C	0	2

* 0 No rusting (control)
1 Very slight rusting
2 More rusting
3 Most rusting

Effect of Temperature on Tooling Material

The data as shown in Table 3 indicate that the rusting of metal tools increases with an increase in temperature when 85% R.H. is used. The rusting appears to be greatest with S5 and least with DM tooling.

Effect of Wet Powder on Tooling Material

The data as presented in Table 4 indicate that the wet powder of REV 6000-A(SS) causes rusting to the tooling material

TABLE 3

EFFECT OF TEMPERATURE ON TOOLING
(2 Hour Observation)

Tooling Material	Rusting (Rating*)	
	22°C/85%RH	35°C/85%RH
D2	1	2
D3	1	2
DM	0	2
S5	1	3
440C	1	2

* 0 No rusting (control)
1 Very slight rusting
2 More rusting
3 Most rusting

due to the ionization of hydrochloride salt in the presence of moisture.

Degree of Rusting on Various Metal Alloys

The results as summarized in Tables 2, 3, and 4 indicate the order of rusting by the various metal alloys as follows:

$$DM < 440C < D2 < D3 \text{ and } S5$$

The tool compositions (Table 1) indicate that the DM has more nickel than the other tooling materials. Nickel has been

TABLE 4

EFFECT OF WET POWDER (10% WATER) ON TOOLING MATERIAL
22°C/30% R.H.

Tooling Material	Rusting (Rating*)	
	2 Hours	24 Hours
D2	2	3
D3	3	3
DM	1	2
S5	2	3
440C	2	3

* 0 No rusting (control)
1 Very slight rusting
2 More rusting
3 Most rusting

reported by Shotton and Ridgway[3] to withstand cold dilute hydrochloric acid up to 15% concentration. These researchers also found that the alloy rich in nickel has good resistance to acid salts and fatty acids. Thus, the development scientist must optimize the choice of tooling based on mechanical properties, chemical resistance and economics. Such screening of tooling material can be incorporated in preformulation testing.

CONCLUSION

REV 6000-A(SS) releases hydrogen chloride under the experimental conditions studied. The liberated hydrogen chloride appears to be responsible for the rusting of tooling materials. Tooling materials rich in nickel content have the least rusting. High humidity also influences the degree of rusting in the presence of labile hydrochloride salts such as REV 6000-A(SS). Based on these conclusions, the rusting of tooling can be prevented by controlling humidity, temperature and by using tooling material rich in nickel alloy.

REFERENCES

1 IPT Standard Specification for Tableting Tools, Academy of Pharmaceutical Sciences, 1971, p. 1.

2 Thomas Engineering, Inc., File Facts 8 and 8B.

3 E. Shotton and K. Ridgway, "Materials of Construction," Physical Pharmaceutics, Clarendon Press, Oxford, 1974, pp. 365-367.